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## EXCHANGE-BIASED NiO-Co NANOFACETED BILAYERS GROWN ON MgO (110)

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### ABSTRACT

We have sputter-grown self-organised faceted NiO-Co epitaxial bilayers on MgO(110). Due to very close lattice parameters, NiO adopts the same NaCl crystallographic structure as the substrate but it minimises its surface energy growing in a stripe-shaped morphology elongated along [001] MgO direction. The Co layers then deposited on NiO adopt a fcc structure. They consist of a set of connected nanowires whose height is about 50 Å, length is near to 1 µm and lateral periodicity  $\approx 100$  Å. Magnetic properties of the Co layers were investigated by magneto-optical Kerr effect from 10 K to room temperature. They are dominated by a strong shape-induced uniaxial anisotropy and exchange coupling with the antiferromagnetic underlayer. Magnetisation loops recorded along the easy axis exhibit a perfect squareness and switch in a field range smaller than 10 Oe. Transverse measurements indicate that switching occurs by domain nucleation and/or domain wall propagation. On the contrary, close to the [110] hard axis, magnetic switching occurs by coherent rotation. The bi-stable Co magnetisation state along its easy axis has been used for ordering the NiO spins configuration from room temperature to 10 K. Sign and value of exchange bias induced by such a thermal treatment can be modulated thanks to a wide magnetocrystalline or local exchange path energies distributions.

### INTRODUCTION

Exchange coupling between a ferromagnet (F) and an antiferromagnet (AF) was discovered more than 40 years ago by Meiklejohn and Bean [1]. This phenomenon has become a great challenge in thin magnetic layers community since it has potential applications in magnetoresistive devices such as spin-valves and tunnel junctions. The main effect induced by this coupling is a typical shift of the F hysteresis loop called exchange bias  $H_E$  and an increase of its coercive field. Ten years ago the first models predicting coherent exchange bias values were proposed by Mauri [2] and by Malozemoff [3]. Both of them point out the main rule played by domain walls in the AF and predict a  $\sqrt{AK}$  dependence of  $H_E$  where A is the AF stiffness and K its anisotropy constant. These models do not only imply that the AF is completely organised after a field cooling through its Néel temperature but the exchange bias sign cannot change either when temperature is decreased further. Exchange bias is expected to follow a  $\sqrt{K(T)}$  dependence. Stiles and Mc Michael [4] have recently proposed a model for polycrystalline samples in which AF grains are divided into two groups according to the  $\epsilon = J_c / \sqrt{AK}$  ratio where  $J_c$  is the average direct coupling between the AF and the F. Grains with low  $\epsilon$  contribute to the exchange bias whereas those with high  $\epsilon$  can induce coercive field. This theory is coherent with experimental studies made by S. Soeya et al in NiO/NiFe [5] bilayers who report that many "local domain paths" between  $T_N(\text{NiO})$  and room temperature are involved in the coupling. These paths can be "activated" reducing temperature. A better understanding of the mechanism of exchange anisotropy requires

AF / F systems as ideal as possible [6][7]. Namely, systems with controlled crystalline coherence of the AF / F interface and reduced dimension like ultra-thin films or nanostructured materials. Efforts have been done to obtain epitaxial NiO-Co bilayers by our group and others like Lai et al. [8] for [001] or [111] NiO-Co bilayers. Defect-free epitaxial systems are supposed to have perfectly flat interfaces over large distances. This is very important for exchange coupling fundamental studies since the spin-ordering in an AF changes every atomic plane. However, real systems like the above-mentioned epitaxial NiO-Co bilayers present roughness features over distances smaller than the usual F and AF domain size, which makes the interpretation of their magnetic behaviour more difficult. This issue may be solved using nanoscale-patterned bilayers. It has been demonstrated, for example, that sub micron scale patterning of continuous layers [9] involved in MTJ's leads to single-domain behaviour. The standard way for obtaining such patterned nano-objects is based on electron beam lithography and requires several steps of fabrication. An alternative way to obtain wire-like aspect ratios for patterned elements is self-organised growth or other structuration methods like the so-called atomic saw [10]. These techniques do not require lithography. Well controlled self-organised growth is expected to lead to the smallest spatial resolution of nanostructures. This way is hardly investigated and we present in this paper our recent results on exchange-biased cobalt nanowires grown on faceted epitaxial nickel oxide layers. MgO (110) single-crystal substrates were used to favour the anisotropic growth of NiO. The NiO top surface presents a roof-like morphology and the Co layers deposited on NiO form an array of connected nanowires. We demonstrate that such nanostructures exhibit a strong uniaxial shape anisotropy combined with exchange bias.

## EXPERIMENTAL DETAILS

Samples were grown in a Plassys MPU 600S ultra high vacuum sputter system on epi-polished MgO (110) substrates. Substrates were first annealed for 1 hour at 900°C with a base pressure better than  $5 \times 10^{-8}$  mB. NiO was sputtered from a facing target magnetron with a deposition rate 0.19 Å/s on the MgO substrates heated at 800°C. In order to have the correct Ni:O stoichiometry, a mixture Ar-O<sub>2</sub> was used for NiO sputtering at  $P = 5.6 \times 10^{-3}$  mB. The NiO thickness of the samples presented in this study ranged from 335 to 1000 Å. A 40 to 160 Å -thick Co layer was deposited at 350°C with a standard magnetron at 0.15 Å/s. In order to organise the spins of the NiO layer relatively to the magnetisation of the Co layer, a 300 Oe field cooling of the as-deposited bilayer was performed in the chamber. In a last step, samples were capped by a 20 Å Mo layer before air exposure. In-situ Reflection High Energy Electron Diffraction (RHEED) was performed at all steps of the growth (bare MgO substrates, NiO layer, Co film) with a 20kV STAIB set-up. After the growth, the samples surfaces were investigated by Atomic Force Microscopy with a Nanoscope III microscope in tapping mode. For Transmission Electron Microscopy (TEM) or High Resolution Electron Microscopy (HREM) studies, the samples were prepared both in plane view (along MgO [110] zone axis) to check the in-plane epitaxial growth and film morphology, and in cross section to observe the stacking sequence along the MgO [001] zone axis. Finally, the magnetic properties were studied at room temperature by Magneto-Optic Kerr Effect (MOKE) with a polarised He:Ne laser ( $\lambda = 6328$  Å).

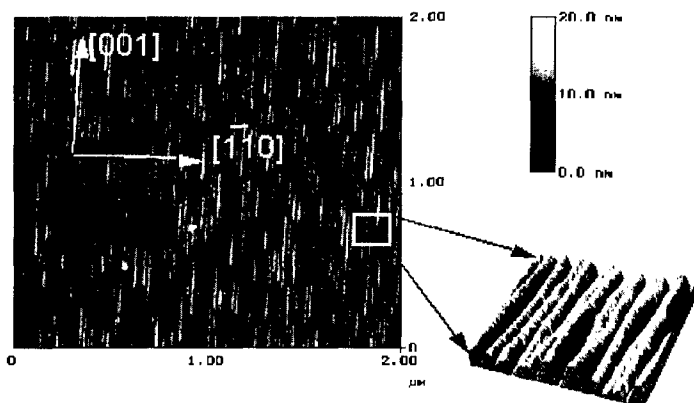


Figure 1: AFM scan of a MgO(110) / NiO(1000 Å) / Co(80 Å) / Mo(30 Å) sample over a  $2 \times 2 \mu\text{m}^2$  area with enlarged  $0.2 \times 0.2 \mu\text{m}^2$  view on the right

## STRUCTURAL PROPERTIES

RHEED patterns, AFM scans and TEM and HREM experiments were carried out on these samples. Structural results are detailed in an other symposium [11]. Let us just point out that all these experiments confirm the NiO self-organised growth. As it can be observed in Fig 1, AFM images reveal a stripe-shaped surface elongated along [001] with symmetrical slopes of about  $45^\circ$  in the [-110] direction. The length of these anisotropic structures is of the order of  $1 \mu\text{m}$  while their width depends on the NiO film thickness. The nanostripes width is close to 80 Å for a 335 Å thick film and reaches 125 Å for a 1000 Å thick NiO layer. Quantitative AFM analysis gives a width distribution of the wires of  $\pm 20$  Å.

## MAGNETIC PROPERTIES

Magnetic properties of NiO/Co bilayers were investigated at room temperature with a magnetic field applied in the plane of the layers. They reveal a strong uniaxial anisotropy along [001] induced by the shape of the Co stripes. Longitudinal and transverse Kerr effect loops along the easy direction [001] and the hard direction [-110] are presented in Fig. 2. Along [001], they have a perfect squareness with a coercive field between 140 and 1500 Oe depending on Co layer thickness. The magnetisation loops along [001] exhibit a very sharp edge and switch in less than 10 Oe despite the Co wires shape distribution; this collective switching implies a coupling between the Co stripes indicative of a contact between each nanowire as observed by HREM. The magnetisation loops measured along [-110], therefore perpendicular to the stripes are characteristic of a hard axis. For all samples, the magnetic field required to saturate the magnetisation in this direction is at least 2200 Oe (see Table I). Transverse Kerr effect loops were also recorded, they evidence a magnetisation switching by coherent rotation if the magnetic field is applied along the [-110] hard axis. Along the easy [001] axis, the small transverse signal is indicative of a magnetisation switching dominated by domain nucleation and/or domain wall propagation.

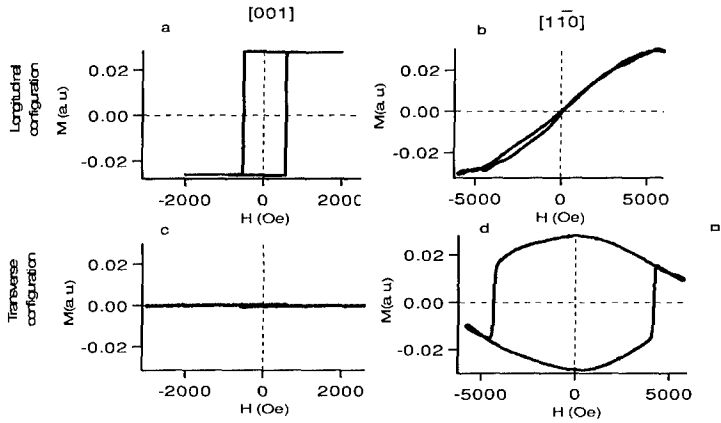


Figure 2: Kerr effect loops in longitudinal (a,b) and transverse (c,d) in-plane configuration recorded along the [001] easy axis (a,c) and the [-110] hard axis (b,d). The same scale is used for the four magnetisation loops

This explains why the easy axis coercive field is lower than the hard axis saturation field contrary to the commonly observed behaviour of uniaxial single domain particles. From the value of the saturation field along the [-110] hard axis, we estimate an uniaxial anisotropy constant of  $1.4 \times 10^6 \text{ erg/cm}^3$  for 160 Å of Co deposited on 335 Å of NiO and of  $5.6 \times 10^6 \text{ erg/cm}^3$  for 40 Å of Co deposited on 335 Å of NiO. This last value is larger than the Co hcp magnetocrystalline anisotropy along the c-axis. Therefore, we assume that the anisotropy observed in our samples is mainly due to their nanowire morphology. This is corroborated by the simple calculation of the demagnetising energy of an infinite Co cylinder which is  $\pi M_{\text{Sat}}^2 = 6.16 \times 10^6 \text{ erg/cm}^3$  and its saturation field is close to  $\pi M_{\text{Sat}} = 8800 \text{ Oe}$ . Such values are almost reached for our very thin Co layer samples, confirming the strong influence of the layer morphology on the Co magnetic anisotropy. The shape anisotropy induced by the roof morphology of the cobalt layer induces a bi-stable state of the Co magnetisation. Indeed, at the remanent state only two fully saturated configurations along its easy axis are allowed. This property has been used to modify the exchange bias value according to temperature. Cooling a MgO(110)/NiO(335 Å)/Co(80 Å)/Mo sample from 300°C to room temperature in a 300 Oe field along the [001] direction induces a -25 Oe exchange bias. This sample was then cut into two pieces. The first one has been cooled from room temperature to 10 K maintaining its magnetisation direction. The second one received the same thermal treatment with its magnetisation aligned along its other stable state which is [00-1]. Hysteresis loops along [001] direction were then recorded warming the samples up to room temperature. Exchange bias values measured and presented in Fig. 3. When the cobalt layer magnetisation is not reversed, exchange bias value is minimum at 10 K and reaches -60 Oe. On the contrary, when the cobalt magnetisation is reversed at room temperature, we can notice a sign change of the hysteresis loop shift which is equal to 20 Oe at 10 K. In both cases the phenomenon is reversible since the exchange bias progressively reaches its room temperature value during the warm up.

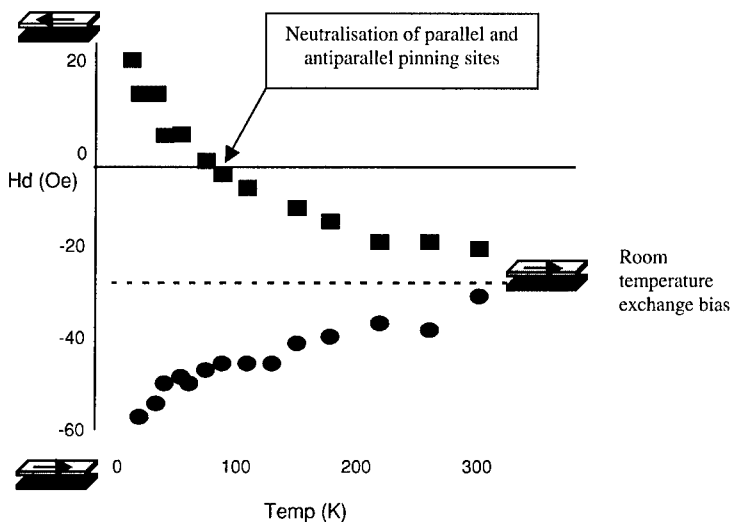


Figure 3: exchange bias temperature dependence according to the cobalt layer magnetisation direction

## DISCUSSION

This phenomenon can be explained extending the Stiles and Mc Michael [4] model to our epitaxial samples. Assuming a wide distribution of the  $J_c / \sqrt{AK}$  rate, the antiferromagnetic layer can be divided in local domain paths which contribute to the exchange bias or to irreversible motion of domain wall within NiO layer. Cooling down the sample, AF magnetocrystalline anisotropy will increase and reach a temperature at which a given domain path will become “active”. This temperature is called local blocking temperature. The initial field cooling has organised all the domain paths whose blocking temperatures are comprised in the range [300°K - 525°K].

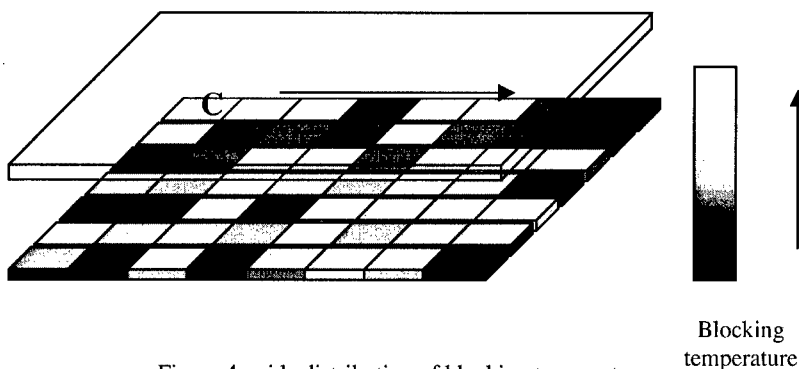


Figure 4: wide distribution of blocking temperature

During the second cooling, low blocking temperature paths have been activated in the same or in the opposite direction to the initial cooling according to cobalt magnetisation direction. Warming up the sample will progressively disable domain paths and reach the same exchange bias value at room temperature induced by high blocking temperature domain paths. Note that an equilibrium between parallel and antiparallel domain paths is obtained near 80 K when the cobalt magnetisation is reversed after the initial cooling. This is mentioned in Fig.3 as a neutralisation temperature. Such distribution has been previously observed by Soeya et al. [5] on polycrystalline NiO/NiFe samples. They conclude that higher blocking temperature density is very close to the global blocking temperature near 215°C. In our samples the distribution is wider and spreads down to 10 K at least. The main teaching of this experiment is that models which describe the NiO magnetic structure as completely frozen just under its Néel temperature are not fully realistic.  $H_E$  value and the NiO magnetic organisation depend on temperature and on the cobalt magnetisation orientation (if the applied magnetic field is low enough to prevent AF spin-flop).

## CONCLUSION

We have prepared exchange-biased NiO-Co bilayers by a combination of facing-target and standard magnetron sputtering. The growth of NiO on MgO(110) leads to a roofed surface with (100) and (010) nanofacets. The Co overlayers adopt this morphology and can be considered as connected nanowires with a strong uniaxial shape-induced anisotropy. The bi-stable Co magnetisation state along its easy axis has been used for ordering the NiO spins configuration from Néel temperature to 10°K. Sign and value of exchange bias induced by a thermal treatment can be modulated thanks to a wide magnetocrystalline or local path blocking temperature distributions.

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